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**by Jared M. Gardner, James P. Wolbert, Larry R. Holmes, Jr., and
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ARL-RP-458

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*A reprint from the 2012 SAMPE Conference Proceedings,
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ABSTRACT

Because of the extensive costs associated with building composite laminates, the U.S. Army is exploring approaches to reducing process steps associated with hybridized structures. One area of focus is in surface preparation for secondary bonding. An important quality of an effective surface preparation method is its ability to consistently provide clean and highly chemically active substrates for bonding. In this report, we examine peel ply surface preparation methods for secondary bonding of composite substrates using FM 94K epoxy film adhesive by mechanical testing, elemental surface analysis, and high-resolution imaging of failure surfaces. Woven S2 fiberglass infused with SC-15 toughened epoxy resin composites laminates, were fabricated by the Vacuum Assisted Resin Transfer Molding process. Tool-side surfaces of the composites were prepared using nine commercial off-the-shelf peel plies, including dry and impregnated forms. Two preparation methods for bonding were investigated: peel ply only-prepared surfaces and peel ply in combination with grit blast-prepared surfaces. This report covers testing results concluded by a quantitative and qualitative analysis of the comparative effects of the preparation methods, technical challenges and influential factors controlling peel ply effectiveness in bond performance, and recommendations for future work.

1. INTRODUCTION

Lightweight composite structures offer significant long-term cost-saving opportunities for the U.S. Army as replacements for traditional metal structures. However, high-strength economical bonding processes must be developed to join composites in order for them to be viable replacements. The Army is exploring approaches, also under evaluation in the commercial aerospace industry, to reduce the process steps associated with composite bonding [1]. A potential single-step process for preparing composite surfaces uses removable fabrics known as peel plies, which are cured to the composite surface during fabrication. Traditionally, peel plies have not been a reliable media for creating optimal bonding surfaces. However, pre-impregnated versions, known as wet peel plies, are a fairly recent innovation that shows significantly improved potential, though at a greater cost compared to their traditional dry fabric counterparts, in creating excellent composite bonding surfaces. Still, reducing the processing steps would reduce overall cost and shorten production lead times of composite systems for the Army in applications such as composite armor and structural vehicle components.

Typically, dry peel ply fabrics have been used as a protective layer for the composite surface after fabrication. The peel ply is removed prior to surface preparation methods for secondary bonding, which include mechanical abrasion, chemical treatments, cleaning, and inspection. The success or failure of a peel ply to create a bondable surface is defined by its ability to

consistently provide clean and highly chemically active substrates. Successful removal of a peel ply requires fracture within the resin matrix between the peel ply and reinforcement fibers, and complete removal of the peel ply fabric without residual fiber transfer. Undesirable modes of removal include interfacial fracture between the peel ply and the matrix, peel ply fracture, and inter-laminar failure within the composite [2]. Transfer of release agents used to facilitate their removal has been widely shown to have significant negative influence on bond performance [3]. Virtually all previous studies that focus on the effects of peel plies on composite bonding conclude that there is no universally acceptable peel ply.

Most current commercially available peel plies are dry fabrics with or without a release coating applied. Typical yarn types are nylon, polyester, and fiberglass in various weave styles. Common release coatings include silicone, polyamide, and polytetrafluoroethylene (PTFE). Non-coated fabrics are typically polyester because it is highly inert. Several fabric characteristics reported to be critical to successful release include yarn type, weave style, and release coating. However, previous studies show varying conclusions regarding the influence of peel ply characteristics on bond performance [2-5]. Developmental research of pre-impregnated peel plies by the Henkel Corp. (Rocky Hill, CT) highlights the combined influence of weave characteristics on wetting/encapsulation of the fabric, and resin toughness, significantly affecting the release of the fabric from the surface [6]. These studies are focused primarily on peel plies used for autoclave processing of pre-impregnated fiber composites.

In this work, we examine surface preparation methods for secondary bonding of composite substrates using FM[®] 94K (Cytec, Havre de Grace, MD) epoxy film adhesive. Laminates using the U.S Army legacy composite system consisting of plain-woven S2 fiberglass (BGF, Greensboro, NC) and SC-15 epoxy resin (Applied Poleramic Inc., Benicia, CA) were prepared by the Vacuum Assisted Resin Transfer Molding (VARTM) process. Two methods were used for tool-side surface preparation: various commercial off-the-shelf (COTS) peel plies, and peel ply in combination with grit blast-prepared surfaces. We also compared nonprepared virgin surfaces, and grit blast only-prepared surfaces. One dry peel ply fabric from Richmond Aerovac (Santa Fe Springs, CA), seven products manufactured by Airtech International Inc. (Huntington Beach, CA), and one pre-impregnated peel ply are examined. Henkel's Wet Peel Ply (WPP), Hysol[®] EA 9896[™]WPP was an experimental epoxy pre-impregnated peel ply system at the time of this work. The U.S. Army Research Laboratory's (ARL) current preparation method incorporates the use of a dry polyamide coated nylon peel ply fabric followed by a grit blasting process. The following tests have been conducted in ambient conditions. Lap shear tests were conducted to establish initial bond strengths. Floating roller peel tests were used to observe the mode of failure at the composite surface. Elemental surface compositions were determined by x-ray photoelectron spectroscopy (XPS). High resolution images of the prepared composite surfaces are examined by scanning electron microscopy (SEM). In addition, qualitative assessments regarding the use and performance of the peel ply fabrics are made.

2. EXPERIMENTATION

2.1 Materials

The S2 fiberglass is a 240-style 5 x 5 plain weave fabric with an aerial weight of 813 g/m² (24 oz/yd²) and 463 epoxy compatible sizing [7]. SC-15 is a low viscosity two-phase toughened epoxy resin matrix designed for VARTM processes [8]. The rheological properties of SC-15

have previously been investigated by ARL and are known to be acceptable for resin infusion processes [9]. The typical fiber volume fraction of a cured laminate for this material system fabricated by the VARTM process ranges between 49 - 52 % with a typical void volume of ~ 1 %, also previously established. Nominal cured laminate thicknesses were 6.35 mm (0.25 in).

The cure schedule used for SC-15 was previously established based on experimental work at ARL (table 1) and varies from the manufacturer's recommended cure schedule [8].

Table 1: ARL cure schedule for VARTM processing of SC-15 epoxy.

Acclimate	Ramp 1	Ramp 2	Hold 1	Ramp 3	Hold 2	Cool
35 °C	0.3 °C/min to 52 °C	to 60 °C	45 min (to gel)	2.2 °C/min to 122 °C	180 min	Hold vacuum pressure 95 kPa (14 psi) 12 hr during cool to ambient

Tool-side surfaces of the composites were prepared using COTS peel ply fabrics, including eight dry and one experimental impregnated form. The peel plies investigated in this work are listed, and the related technical data as supplied by the manufacturers is shown in table 2 [10-12].

Table 2: Peel ply materials examined in this study.

Peel Ply	Yarn Material	Coating Type ^a	Construction ^a End/dm x Pick/dm (End/in x Pick/in)	Weight g/m ² (oz/yd ²)	Thickness ^a mm (in)	Manufacturer
Bleeder Lease [®] A	Nylon	Silicone	158 x 158 (40 x 40)	80 (2.34)	0.15 (0.0058)	Airtech International Huntington Beach, CA
Bleeder Lease [®] B	Nylon	Silicone	409 x 342 (104 x 87)	62 (1.83)	0.11 (0.0045)	Airtech International Huntington Beach, CA
Bleeder Lease [®] C	Fiberglass	Silicone	224 x 213 (57 x 54)	299 (8.8)	0.33 (0.013)	Airtech International Huntington Beach, CA
Dahltextx SP-2	Nylon	<i>PTFE</i>	<i>260 x 189</i> <i>(66 x 48)</i>	140 (4.12)	0.22 (0.0087)	Airtech International Huntington Beach, CA
Econo Ply E	Polyester	None	<i>283 x 213</i> <i>(72 x 54)</i>	95 (2.8)	0.15 (0.006)	Airtech International Huntington Beach, CA
Bleeder Lease [®] G	Polyester	Silicone	472 x 252 (120 x 64)	83 (2.45)	0.13 (0.0050)	Airtech International Huntington Beach, CA
Hysol [®] EA 9896 [™] WPP	Nylon (carrier)	Epoxy (45 wt. %)	<i>629 x 394</i> <i>(160 x 100)</i>	122 (3.59)	<i>0.15</i> <i>(0.006)</i>	Henkel Aerospace Bay Point, CA
Richmond A-8888	Nylon	Polyamide	<i>158 x 158</i> <i>(40 x 40)</i>	75 (2.2)	0.11 (0.0045)	Richmond Aerovac Santa Fe Springs, CA
Superlease Blue	Nylon	Silicone	409 x 342 (104 x 87)	62 (1.83)	0.11 (0.0045)	Airtech International Huntington Beach, CA

^a Italicized values were determined experimentally and were not provided by the manufacturer.

All bonding was conducted in a convection oven, using Cytec's FM[®] 94K preimpregnated modified epoxy film adhesive, according to the cure schedule in table 3. FM[®] 94K has an aerial weight of 146 g/m² (4.30 oz/yd²) and polyester knit carrier resulting in a 0.15 mm (0.006 in) cured ply thickness [13].

Table 3: FM[®] 94K epoxy cure schedule as used in this study

Ramp	Hold	Cool
Apply vacuum pressure 95 kPa (14 psi) 2.2 °C/min to 121 °C	60 min	Hold vacuum pressure 95 kPa (14 psi) 12 hr during cool to ambient

2.2 Laminate Fabrication

Composite laminates were constructed using 8 plies of the plain-woven S2 fiberglass fabric in a 0/90 stacking sequence, infused with SC-15 epoxy resin by the VARTM process. Peel ply dimensions were cut oversized by 76.2 mm (3 in) relative to the reinforcement plies allowing for approximately 38.1 mm (1.5 in) extension beyond the part edges.

Laminate layup and curing processes were performed on the surface of an electronically controlled BriskHeat Corp. (Columbus, OH) heat table with insulating blankets placed over the laminates during the curing cycle. The surface of the heat table was sanded using 120-grit sand paper and cleaned with acetone prior to laminate fabrication. Henkel's Frekote[®] 44-NC[™] mold release agent was applied to surface of the heat table, allowing 15 minutes for solvent evaporation between successive coats and 60 minutes prior to panel fabrication [14].

Peel plies were placed directly on the surface of the tool beneath the reinforcement ply stack. The warp direction of the peel ply and reinforcement plies were consistently oriented in the flow direction of the resin infusion process. A primary and secondary (redundant) vacuum bag was used in the fabrication of all laminates, achieving approximately 95 kPa (14 psi) vacuum pressure during processing. Ply stacks were debulked overnight prior to resin infusion. The layup and bagging sequence used for the VARTM process is illustrated in figure 1.

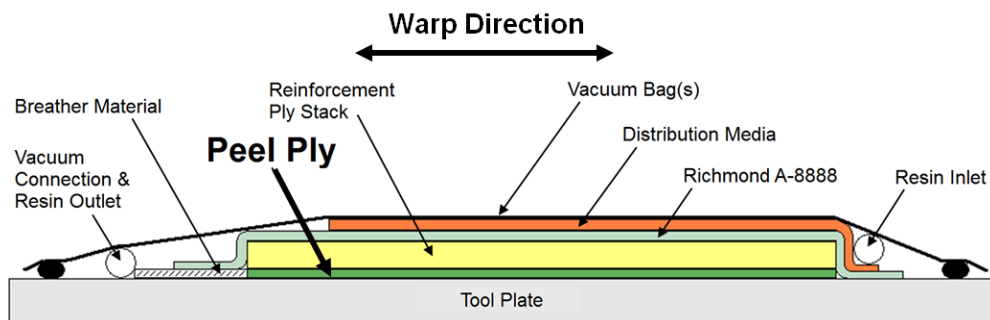


Figure 1: Schematic of the layup and bagging sequence for the VARTM process.

The resin was mixed for two minutes at a moderate rate using alternating mixing patterns then degassed in a vacuum oven for 8 to 10 minutes to remove trapped air. The average resin infusion process required between 15 and 20 minutes for complete wetting to be achieved.

Specimen machining of lap shear and floating roller peel specimens was performed using a Flow International Corp. (Kent, WA) water-jet cutting system. The long direction of the specimens tracked the warp direction of the laminates, corresponding to the infusion flow direction during laminate fabrication. Specimens were immediately rinsed and then dried in an oven at 60 °C (140 °F) for 120 minutes. Peel plies remained on the bonding surfaces of the cured laminates during machining in all cases and were not removed until immediately prior to bonding.

2.3 Analysis of Peel Ply Removal

Simple observations made during the peel ply removal process can provide indicators regarding overall performance, and practicality of use. These observations were documented and a qualitative comparison of the processes was made. The peel plies were stripped from the substrates of two 76.2 mm x 76.2 mm (3 in x 3 in) specimens representing each peel ply, and an assessment of the relative force required for removal was recorded. Additional removal characteristics, such as edge fraying and fiber transfer, were documented during preparation of lap shear coupons. Peeling was initiated using a razor blade, starting at square corner edge, and peeling diagonally away from the initiation point.

2.4 X-Ray Photospectroscopy (XPS) Analysis

A Kratos Ultra165 XPS was used to characterize the near-surface composition of the prepared composite surfaces. Examination of the surface compositions will show differences in makeup and identify any transfer of contaminants which occurred during the removal process. Survey and high resolution scans were made at pass energies of 80 eV and 20 eV to analyze a 1 x 0.5 mm² spot size. The take off angle was 90°, and all spectra were calibrated according to the hydrocarbon peak at 285 eV. A vacuum environment of 2×10^{-9} torr or lower was achieved. Prior to analysis, specimens were oven dried at 60 °C (140 °F) for 120 minutes. Peel plies were removed from the surfaces immediately prior to insertion into the test chamber.

2.5 Scanning Electron Microscope (SEM) Analysis

Fiber transfer or surface damage from the removal process, and surface wetting characteristics were not apparent by visual examination of the prepared surfaces. For closer examination, a Hitachi S-4700 FESEM was used to obtain high resolution images of the specimen surfaces. Imaging primarily was conducted at 5 kV and 40x magnification on the prepared surfaces. The specimens were placed in an oven and dried at 60 °C (140 °F) for 120 minutes. The peel plies were removed from the surfaces immediately prior to sputter coating. A gold/palladium coating was applied to the surfaces using a Denton DESK V – TSC sputter coater (Moorestown, NJ). Sputter conditions were 40 seconds at a sputter set point of 40 and 1×10^{-3} torr pressure.

2.6 Lap Shear Testing

Two series of composite-to-composite lap shear tests were conducted at ambient conditions according to ASTM D5868 – 01 [15]. Series 1 contained peel ply-prepared sample groups and an as-tooled virgin surface sample group. Series 2 contained peel ply followed by a grit blast sample groups and a grit blast-only sample group. Each sample group consisted of six specimens.

Laminates were fabricated to 304.8 mm x 304.8 mm (12 in x 12 in) dimensions and machined into 25.4 mm x 101.6 mm (1 in x 4 in) coupons. Test specimens were bonded using two plies of FM[®] 94K epoxy film adhesive. All bonding was conducted according to the cure schedule shown in table 3. Final bonded joint dimensions were nominally 25.4 mm x 25.4 mm (1 in x 1 in) with a cured adhesive bond thickness of 0.30 mm (0.012 in). The composite surface preparation methods for lap shear testing are summarized in table 4. A schematic of the layup sequence for the lap shear specimens is shown in figure 2.

Table 4: Composite surface preparation methods for lap shear testing.

Sample Group	Peel Ply Removal	Grit Blast (180-grit Al Oxide)	Blow Off (Nitrogen Gas)	Solvent Wipe (IPA)
Series 1 as-tooled	-	-	-	✓
Series 1 peel ply	✓	-	✓	-
Series 2 grit blast	-	✓	✓	✓
Series 2 peel ply	✓	✓	✓	✓

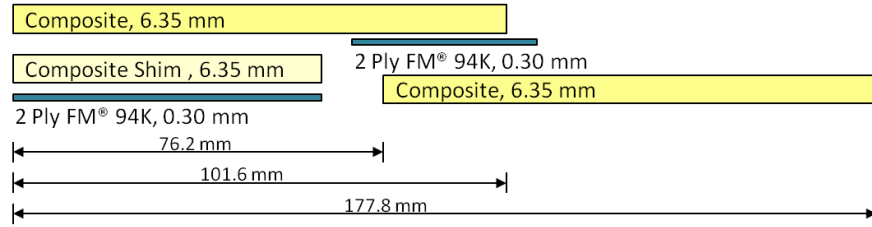


Figure 2: Schematic of the layup sequence for the lap shear specimens.

Testing was performed on an Instron (Norwood, MA) model 1123 electromechanical test frame equipped with a 25-kN load cell. Tests were conducted at a loading rate of 12.7 mm/min (0.5 in/min) with 25.4 mm (1 in) of each sample end held in the grips, resulting in an initial grip separation of 127 mm (5 in). Lap shear strength is calculated using equation 1 [16]:

$$LSS = \frac{P_{max}}{A} \quad , \quad [1]$$

where P_{max} is the load at failure, and A is the bond area of the joint.

2.7 Floating Roller Peel Testing

Two series of composite-to-metal floating roller peel tests were conducted at ambient conditions similarly to the recommendation given by ASTM D3167 – 10 [17]. Each series was prepared separately, and each sample group consisted of six specimens, resulting in a total of twelve specimens for each sample type.

Laminates were fabricated to 356 mm x 508 mm (14 in x 20 in) dimensions and machined into two 152.4 mm x 203 mm (6 in x 8 in) panels. The composite surface preparation methods for floating roller peel testing are summarized in table 5.

Table 5: Composite surface preparation methods for floating roller peel testing.

Sample Group	Peel Ply Removal	Grit Blast (180-grit Al Oxide)	Blow Off (Nitrogen Gas)	Solvent Wipe (IPA)
Series 1 & 2 peel ply	✓	-	✓	-
Series 1 & 2 grit blast	-	✓	✓	✓

The flexible metal adherends were cut from clad aluminum alloy 2024-T3 sheet of 0.508 mm (0.020 in) thickness into 152.4 mm x 254 mm (6 in x 10 in) panels. Two grit blast methods were used in preparing the metal adherends so that a comparison of the methods could be made. Series 1 was prepared by grit blasting only the bonding surface. Series 2 was prepared by grit blasting both surfaces. The metal adherends underwent a spray application of silane treatments immediately after grit blasting to improve the interfacial bonding with the adhesive interlayer. The silane treatments consisted of 99 weight percent 90:10 ethanol:H₂O mixture with an adjusted pH of 4.5 and 1 weight percent of 3-glycidoxypropyltrimethoxysilane (GPS) (Fluka Sigma-Aldrich, St. Louis, MO).

Two plies Flashbreaker[®]2 pressure sensitive tape made by Airtech of 25.4 mm (1.0 in) width and 0.075 mm (0.003 in) thickness were stacked and applied to the surface at the end of the panel as a crack starter. One ply of FM[®] 94K was tacked to the remaining uncovered composite surface. Assembled panels were arranged on aluminum tool plates with the metal adherends in contact with the plates. All bonding was conducted according to the cure schedule shown in table 3. A schematic of the layup sequence for the Floating Roller Peel assemblies is shown in figure 3.

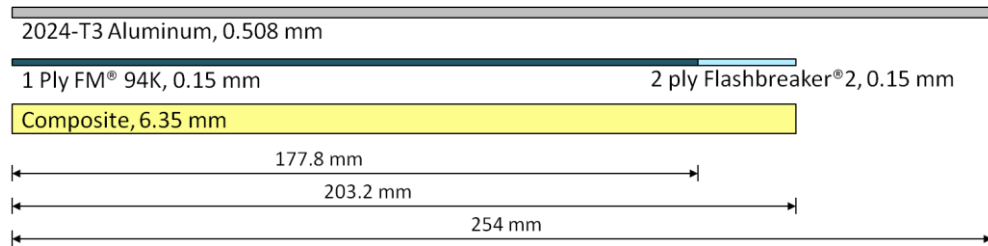


Figure 3: Schematic of the layup sequence for the Floating Roller Peel panel assemblies.

Six 12.7 mm (0.5 in) wide specimens were cut from the center of each of the bonded assemblies. Final bonded joint dimensions were nominally 12.7 mm x 177.8 mm (0.5 in x 7 in) with a cured adhesive bond thickness of 0.15 mm (0.006 in). Specimen widths were measured at locations 63.5 mm (2.5 in), 101.6 mm (4 in), 139.7 mm (5.5 in) from the end of the composite adherend and recorded. The average width of the specimen in this region was used to calculate the load / width of peeling force over 76.2 mm (3 in) length of the bond line experienced during testing. Data recorded over the initial 38.1 mm (1.5 in) and final 38.1 mm (1.5 in) of peel length was disregarded in the analysis.

Testing was performed on an Instron model 1123 electromechanical test frame equipped with a 500-N load cell. Tests were conducted at a loading rate of 152 mm/min (6 in/min) with 25.4 mm (1 in) of the flexible adherend held in the grip of the test fixture.

3. RESULTS

3.1 Analysis of Peel Ply Removal

Table 6 shows a qualitative assessment of the force required for peel ply removal in combination with fabric yarn material, coating type, and fabric weave style. The stripping force required to remove the peel plies from the surface refers to the relative force required to manually peel the fabric away from the surface after substantial initiation. Peel ply fabrics after removal from the substrates are shown in figure 4.

Table 6: Relative stripping force required for peel ply removal listed by material type.

Peel Ply	Yarn Material	Coating Type ^a	Generic Weave Style	Stripping Force Required
Bleeder Lease [®] A	Nylon	Silicone	Open	Medium to High
Bleeder Lease [®] B	Nylon	Silicone	Tight	Medium
Bleeder Lease [®] C	Fiberglass	Silicone	Open	Low
Dahltextx SP-2	Nylon	<i>PTFE</i>	Tight	Medium
Econo Ply E	Polyester	None	Tight	Medium
Bleeder Lease [®] G	Polyester	Silicone	Tight	Low
Hysol [®] EA 9896 [™] WPP	Nylon	Epoxy	Tight	Highest
Richmond A-8888	Nylon	Polyamide	Open	Medium
Superlease Blue	Nylon	Silicone	Tight	Low to Medium

^a Italicized values were determined experimentally and were not provided by the manufacturer.

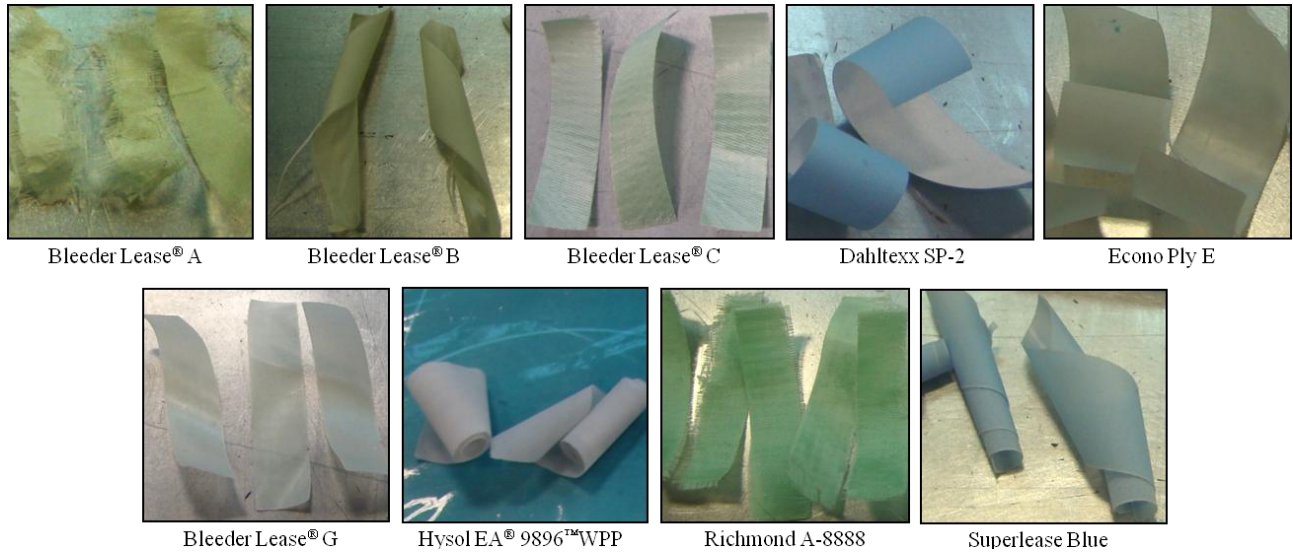


Figure 4: Peel ply fabrics removed from lap shear coupons.

In general, the qualitative assessment regarding peel ply fabric removal in this study is as follows for this composite system:

1. Tight weaves facilitate an easier initiation and more complete removal than open weaves regardless of the type of release coating.
2. Tight weaves require low to moderate stripping force for removal with the exception of the pre-impregnated fabric.
3. Tight weave non-coated polyester requires moderate stripping force for removal.
4. Open weave fabric constructions with the lowest end and pick counts (table 2) have an increased tendency to split along clean cut edges.
5. Silicone coated fiberglass and polyester required the lowest stripping force to remove.
6. The open weave silicone coated nylon fabric showed poor resin wetting/encapsulation.

3.2 X-Ray Photospectroscopy (XPS) Analysis

XPS results showing the elemental near surface composition of the substrates after each surface preparation method are shown in table 7. Elements which indicate contamination are shown in the columns highlighted in red. The surface compositions are shown quantitatively as atomic concentrations detected. The approximate peak binding energy positions are as follows: Si_{2p} ~ 100 eV, Si_{2s} ~ 150 eV, C_{1s} ~ 285 eV, N_{1s} ~ 400 eV, O_{1s} ~ 530 eV, F_{1s} ~ 685 eV. Silicone, chlorine, and fluorine are contaminants that have come into contact with the surfaces.

Table 7: XPS results of peel ply and grit blast surface preparations.

Peel Ply	Yarn Material	Coating Type ^a	Atomic %					
			C 1s	O 1s	N 1s	Si 2s & Si 2p	Cl 1s	F 1s
Bleeder Lease [®] A	Nylon	Silicone	73.0	22.2	1.1	3.8	-	-
Bleeder Lease [®] B	Nylon	Silicone	68.3	19.4	1.5	10.8	-	-
Bleeder Lease [®] C	Fiberglass	Silicone	74.8	21.0	0.6	3.6	-	-
Dahltext SP-2	Nylon	<i>PTFE</i>	74.3	17.2	1.1	-	-	8.0
Econo Ply E	Polyester	None	78.6	21.4	-	-	-	-
Bleeder Lease [®] G	Polyester	Silicone	72.8	22.2	1.1	3.9	-	-
Hysol [®] EA 9896 [™] WPP	Nylon	Epoxy	84.2	12.8	3.0	-	-	-
Grit Blast	NA	NA	78.3	18.6	2.4	0.4	0.3	-
Richmond A-8888	Nylon	Polyamide	76.0	21.5	-	2.5	-	-
Superlease Blue	Nylon	Silicone	74.0	21.8	2.2	2.0	-	-

^a Italicized values were determined experimentally and were not provided by the manufacturer.

Note: NA = not applicable

The XPS results in table 7 show varying surface compositions dependent upon the preparation method used. Presence of Silicone and Fluorine on some specimens is attributable to transfer of

the peel ply release coatings, with the exception of the grit blasted specimen. Silicone and Chlorine detected in the grit blasted specimen are most likely from water lubricated machining operations and residual contamination in the grit blasting equipment.

In general, the XPS analysis of the peel ply-prepared surfaces revealed the following:

1. Transfer of the release agent was detected from all release-coated fabrics.
2. No contaminants were detected from the non-coated polyester fabric or the pre-impregnated fabric.
3. Significant differences in surface composition of the pre-impregnated fabric were noted compared to the dry forms.

3.3 Scanning Electron Microscope (SEM) Analysis

SEM images show surface wetting characteristics at the tool/peel ply interface, surface damage due to peel ply removal or grit blasting, and signs of significant fiber transfer that occurred. Voids at the surface of the composite generally indicate poor surface wetting and are regular in shape. Damage locations are assumed to be irregular in shape and occur near void locations. Signs of potential damage from grit blasting resemble uneven erosion of the surface. Fiber transfer is recognizable in two ways: large fibers that are easily identifiable or small fiber tendrils protruding from the surface.

In general, the SEM analysis of the peel ply-prepared surfaces shown in figure 5 revealed the following:

1. Only the three tight weave nylon fabrics showed complete wetting at the composite/fabric interface (Dahltextx SP-2, Hysol[®] EA 9896[™], and Superlease Blue).
2. Fiber transfer was found on the surface of all composites where incomplete wetting was observed, with the exception of the open weave polyamide-coated nylon fabric (Richmond A-8888).
3. Matrix fracture at the composite/peel ply interface was clearly shown on the pre-impregnated fabric-prepared surface (Hysol[®] EA 9896[™]).
4. Surface damage was observed from the non-coated polyester fabric.
5. Surface damage was observed from the grit blasting process.
6. All peel plies in this work appear to have fractured from the surfaces within the matrix at the composite/fabric interface.

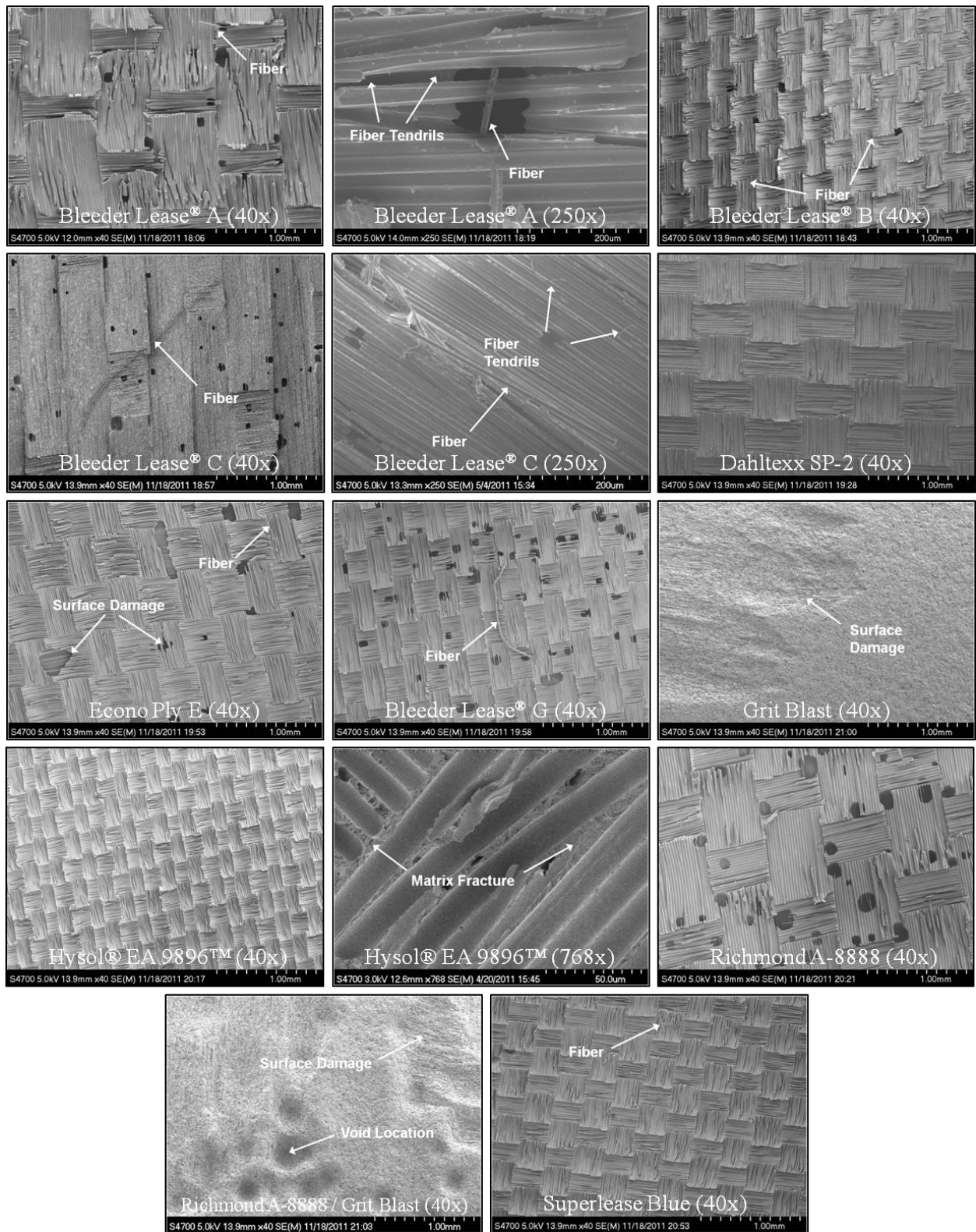


Figure 5: SEM images of peel ply and grit blast prepared composite surfaces.

3.4 Lap Shear Testing

The lap shear strength results for series 1 (peel ply and as-tooled samples) are shown in figure 6. Lap shear strengths show two clear groupings in the results which seem distinguishable by comparing tight and open fabric weave styles. The tight weave fabric preparations had the highest strengths (13.9 MPa to 15.4 MPa) with the exception of Bleeder Lease® B, which had an extremely high concentration of silicone on the surface (table 7). The remainder of the group, which is found to have lower strengths (11.2 MPa to 12.6 MPa), comprises the open weave fabric preparations. The grit-blasted sample was only slightly higher in strength (12.7 MPa) than the open weave-fabric prepared samples. The pre-impregnated fabric preparation (Hysol EA® 9896™) had the highest strength (15.4 MPa) by nearly a factor of 2 over the as-tooled samples, which had the lowest strength (7.8 MPa).

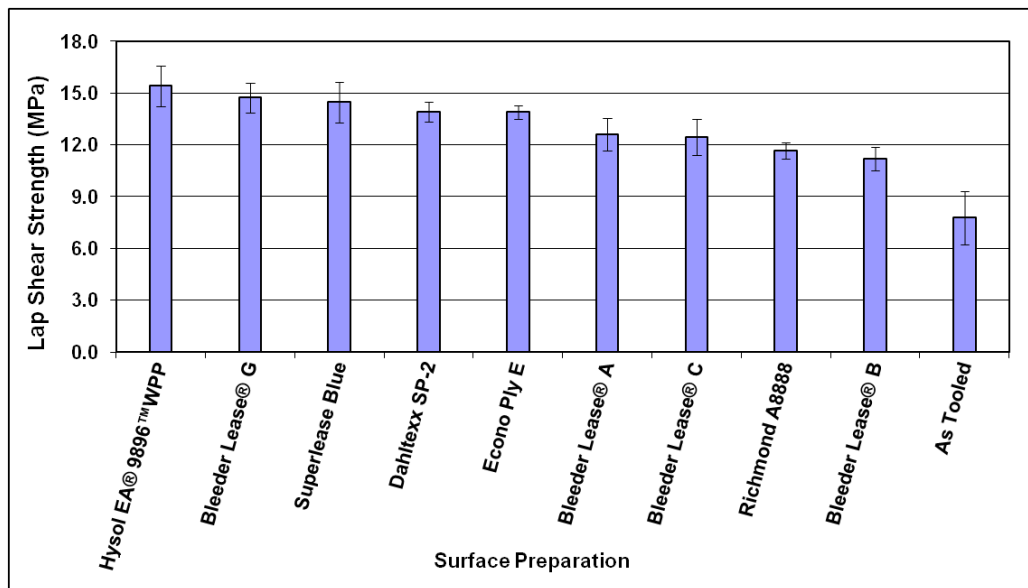


Figure 6: Series 1 lap shear strength results for the peel ply and as-tooled samples.

The results from series 2 (peel ply followed by grit blast) lap shear testing are shown in figure 7. Series 1 (peel ply only) results are also shown for comparison. Most of the lap shear strengths show reduced standard deviations compared to series 1. A tighter grouping of the different preparations is also observed compared to series 1. All sample groups prepared first by peel ply show higher strengths than the grit blast-only prepared sample. Some lap shear strengths increased and some decreased by combining peel ply and grit blast preparations. The increases are most likely due to the removal of fiber and release agent transfer after grit blasting. Decreases in strength are not significant and are likely attributable to the small sample size.

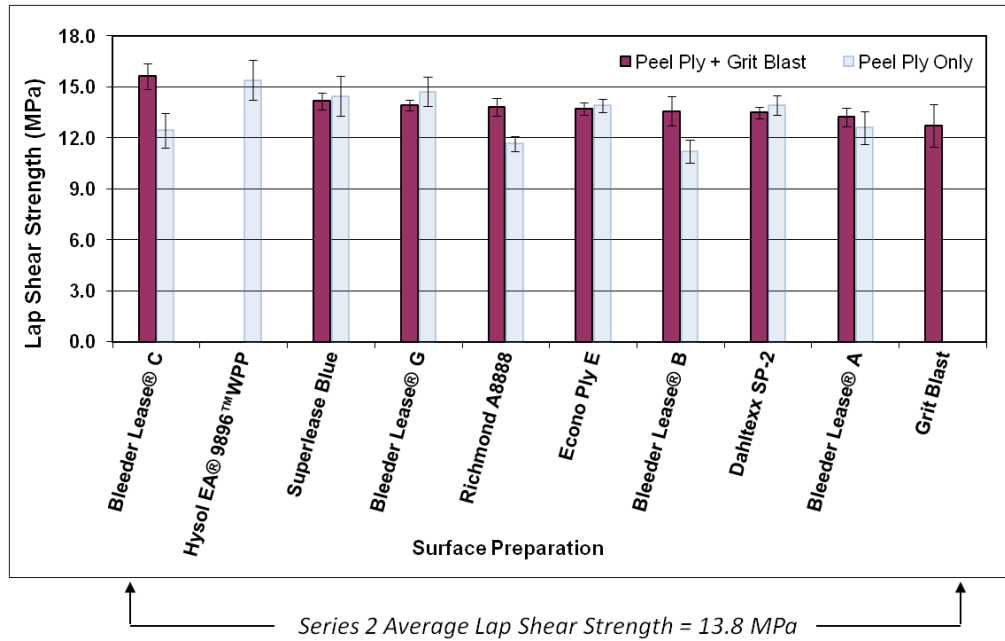


Figure 7: Series 2 lap shear strength results for the peel ply followed by grit blast and grit blast-only samples. Series 1 peel ply-only results are also shown for comparison.

The failure modes observed for each sample group are shown in table 8. The open weave fabric preparations showed mixed modes of failure in the peel ply-only prepared samples. The tight weave fabric preparations showed cohesive failures with the exception of Bleeder Lease® B. The as-tooled specimen failures were completely adhesive. All grit blast-prepared specimens showed cohesive failure.

Table 8: Primary failure modes observed in lap shear testing

Surface Preparation	Generic Weave Style	Peel Ply (Series 1)	Grit Blast (Series 2)
Bleeder Lease® A	Open	Mixed	Cohesive
Bleeder Lease® B	Tight	Mixed	Cohesive
Bleeder Lease® C	Open	Mixed	Cohesive
As-Tooled	Tight	Adhesive	-
Dahltexx SP-2	Tight	Cohesive	Cohesive
Econo Ply E	Tight	Cohesive	Cohesive
Bleeder Lease® G	Tight	Cohesive	Cohesive
Hysol EA® 9896™WPP	Tight	Cohesive	-
Grit Blast	-	-	Cohesive
Richmond A8888	Open	Mixed	Cohesive
Superlease Blue	Tight	Cohesive	Cohesive

3.5 Floating Roller Peel Testing

Mode of failure is the dominant assessment for this test, more so than failing load. The failure modes observed during floating roller peel testing are shown in figure 8. Cohesive failure in the adhesive at the composite surface was observed for Econo Ply E, grit blast, Hysol[®] EA 9896[™], and Superlease Blue specimens. The failure mode for Bleeder Lease[®] A and Bleeder Lease[®] B was predominantly adhesive at the composite surface with a small percentage of the failure occurring cohesively. Bleeder Lease[®] C, Dahltexx SP-2, Bleeder Lease[®] G, and Richmond A-8888 peel plies produced nearly complete adhesive failures at the composite surface.

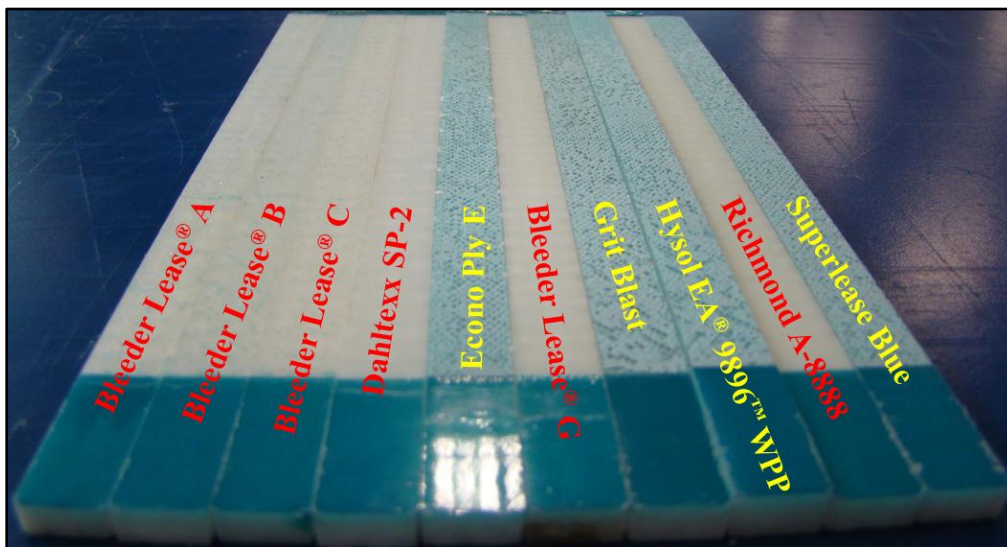


Figure 8: Image of the composite failure surfaces from floating roller peel testing. Red font indicates primarily adhesive failures. Yellow font indicates cohesive failures.

The angle of peel must be controlled for all specimens tested and is critical for making direct comparisons between samples. To make a valid comparison, the flexible adherend must bend over the mandrel and not at some irregular angle; an irregular angle could result in the angle of peel not being identical [17]. Data generated during Floating Roller Peel testing showed a valid comparison between Econo Ply E, grit blast, Hysol[®] EA 9896[™], and Superlease Blue preparations. These samples displayed the proper angle of peel in the test fixture. In addition, these samples produced cohesive failures and significant plastic deformation was observed in the flexible metal adherends. All other surface preparation methods that were tested produced non-valid strength results for this comparison because of to improper angle of peel in the test fixture. These samples generated negative load data due to adhesive failures and did not produce any significant plastic deformation in the flexible metal adherends.

Testing results for the samples where cohesive failures were observed are shown in figure 9. Large differences in values between series 1 and 2 for Econo Ply E, Hysol EA[®] 9896[™]WPP, and Superlease Blue are observed. The grit-blasted specimens showed the lowest standard deviation of the combined results. It is difficult to conclusively determine if the two grit-blasting procedures used on the flexible metal adherends had a significant effect on the results. However three sample types showed significant increases in strength in series 2 versus series 1.

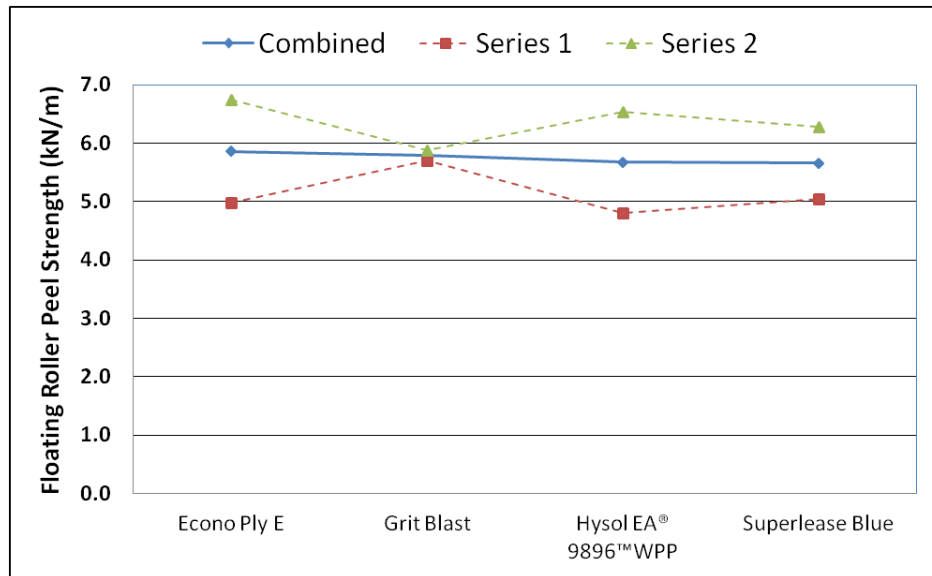


Figure 9: Floating Roller Peel strength results for the peel ply and grit blast-prepared surfaces from which cohesive bond failure modes were observed. Results from series 1, series 2, and the combined averages of all specimens are shown.

4. CONCLUSIONS

Composite surface preparation of S2 fiberglass and SC-15 epoxy composites using commercial peel ply fabrics was evaluated and compared to current grit blast preparation methods. Three peel ply preparation methods (Econo Ply E, Hysol® EA 9896™WPP, and Superlease Blue) showed desirable cohesive failures in peel testing. Of these materials, only Hysol® EA 9896™WPP showed desirable characteristics in all tests, bond strength, mode of failure, and fabric removal. Grit blast surface preparations showed desirable failures in all tests, although peel ply combined with grit blasting improves strengths over grit blasting only. This approach most likely reduces variability in bond performance, provided a clean surface is generated after the peel ply removal and grit blasting. Transfer of release agents was discovered on most of the peel ply-prepared surfaces with the exceptions of Hysol® EA 9896™ pre-impregnated nylon fabric and Econo Ply E non-coated polyester fabric. However, XPS and lap shear results for Hysol® EA 9896™WPP indicate significant changes in surface chemistry most likely because of co-mingling of the matrix resins of the system and peel ply. The chemistry of the pre-impregnating resins for wet-peel-ply fabrics may be tunable for use with specific material systems in order to meet the high-durability bonding requirements of the United States Army. This is a key requirement that must be met and could allow for their use in composite bonding for Army applications, therefore allowing a reduction in process steps. In general, all peel ply fabrics most likely influence surface chemistry. However, it is unknown how these changes will affect important bond properties such as impact and durability in military composite structures. Dry peel ply fabrics are not a desirable single-step surface preparation method for the composite surfaces examined in this study. However, their use is justified, provided a sufficiently clean surface can be obtained by grit blasting prior to bonding.

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